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(54) Title of the Invention: **Absorbent Material**

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## **SPECIFICATION**

### **1. Title of the Invention**

Absorbent Material

### **2. Claims**

An absorbent material comprising an acrylic acid superabsorbent resin monomer solution applied to and polymerized on a fiber web, characterized in that

the fiber web comprises at least two fiber layers with different fiber base structural densities, and the density of the superabsorbent resin is varied in the widthwise direction of the fiber layers.

### **3. Detailed Description of the Invention**

Field of Industrial Application

The present invention relates to an absorbent material that comprises superabsorbent resin and that is suitable for disposable diapers and the like which rapidly absorb large amounts of urine, for example, without leaking, and in particular relates to a thin, compact absorbent material with better performance comprising a superabsorbent resin capable of more efficiently exploiting latent absorption capacity and rapidly absorbing large amounts of moisture.

#### **Prior Art**

Absorbent materials featuring the use of superabsorbent resins have recently been proposed for use as disposable sanitary materials such as disposable diapers. Such absorbent materials will be discussed below using the example of disposable diapers.

As illustrated in Figure 8, disposable diapers usually comprise a moisture-permeable top sheet 1 on the side in contact with the crotch area of infants, invalids, elderly individuals, or the like, a liquid-absorbing retention layer 2 located between the above layer and the following layer, and a moisture-impermeable backing sheet 3 on the outside. The liquid-absorbing retention layer 2 conventionally comprises a mixture of milled wood pulp fibers and microparticulate superabsorbent resin, which is used either as such or after being formed into layers, but the absorption capacity is currently unsatisfactory. Methods have thus been proposed for obtaining the following effects by improving the internal structure of absorbent materials and by providing

a concentration (density) gradient in the widthwise direction of the distribution of the superabsorbent resin in the absorbent material, such as by making the distribution looser on the side in contact with the body and denser on the side away from the body.

1) The proposals prevent the formation of what are referred to as gel blocks, which interfere with the permeation of urine into the inner layers after a layer is formed by adjacent resin swollen with absorbed urine.

2) Portions with low resin density serve as reservoirs for large amounts of excreted urine until the urine can be absorbed by the superabsorbent resin which has an inherently slower rate of absorption.

3) The proposals prevent discomfort resulting from direct contact on the skin by sticky urine-soaked resin which has become expanded.

Figure 9 illustrates an example of a concentration (density) gradient provided in the widthwise direction of the distribution of the superabsorbent resin in an absorbent material, where a mixture of a superabsorbent resin 4 and wood pulp fibers 5 mixed in varying proportions is laminated in layers.

However, it is extremely difficult to obtain an absorbent material with a concentration (density) gradient in the widthwise direction of the superabsorbent resin distribution in absorbent materials in which the superabsorbent resin is formed by polymerization after the application of an acrylic acid superabsorbent resin monomer solution to a fiber web, as in the present invention.

#### Problems Which the Invention Is Intended to Solve

An object of the present invention is to provide an absorbent material comprising a superabsorbent resin fixed to a fiber base, with a structure enabling adjustment of the concentration (density) gradient of the superabsorbent resin in the widthwise direction in the absorbent material.

#### Means for Solving the Abovementioned Problems

The inventors perfected the present invention upon discovering that the density of the fiber base constituting a fiber web could be varied to change the amount of monomer solution adhering to the fiber web, and that the concentration (density) of the resin in the widthwise

direction of the absorbent material could be freely adjusted by making use of the changes in the amount of monomer solution that adhered, in response to the density of the fiber base density, to the fiber layers when the monomer solution was applied to a fiber web formed by the lamination of fiber layers with varying fiber base densities.

That is, the absorbent material of the present invention is an absorbent material comprising an acrylic acid super absorbent resin monomer solution applied to and polymerized on a fiber web, characterized in that the fiber web comprises at least two fiber layers with different fiber base structural densities, and the density of the super absorbent resin is varied in the widthwise direction of the fiber layers.

The absorbent material of the present invention can be realized simply by varying the structural density of the fiber base of the individual fiber layers forming the fiber web, so that the density is, starting on the side in contact with the skin, loose to compact or compact to loose, loose to intermediate to compact or compact to intermediate to loose, or if desired, loose to compact to loose or compact to loose to compact.

The density of the fiber base is defined as the surface area ( $\text{cm}^2$ ) of the fiber base contained in the unit volume ( $1 \text{ cm}^3$ ) of the fiber layers. In combinations of fiber bases of the same basis weight and same denier, the lower the bulk the greater the fiber base density. Conversely, the lower the fiber base denier at the same basis weight and same bulk, the greater the fiber base density.

The concentration (density) distribution of the superabsorbent resin in the absorbent material of the present invention is not particularly limited, but as noted above, is preferably looser on the side in contact with the body, and is preferably more compact on the side away from the body, in order to ensure better absorption performance. When the fiber web comprises two layers of fiber web (A) and (B), as illustrated in Figure 1, for example, the density of the two fiber bases should be at least  $5 \text{ (cm}^2\text{)}$  different, where the density of the fiber base F of the fiber layer (A) on the side in contact with the body, which has a looser resin concentration (density) distribution, is  $5 \text{ to } 25 \text{ (cm}^2\text{)}$ , and the density of the fiber base F of the fiber layer (B) on the side away from the body, which has a more compact resin concentration (density) distribution, is  $10 \text{ to } 50 \text{ (cm}^2\text{)}$ . When the fiber web comprises three or more fiber layers, the fiber base density of the fiber layer on the side in contact with the body and the fiber layer farthest from the body should

be the same as the fiber layers (A) and (B) above, and the resin concentration (density) between those two fiber layers should increase from the direction in contact with the body toward the direction away from the body.

The following advantages, among others, can be obtained by making the distribution of the superabsorbent resin in the absorbent material compact on the side in contact with the skin, loose in the intermediate layer, and compact again on the side farthest from the body. A) It will improve the flexibility of the absorbent material, provide a softer hand, improve adhesion on the body, and prevent leakage. B) The intermediate layer with the loose resin distribution will act as a reservoir for the urine, thus allowing large quantities of urine that are eliminated at once to be rapidly taken up into the absorbent material and to be rapidly absorbed into the upper and lower resin layers. C) Although the superabsorbent resin expands after absorbing urine and disrupts the fiber skeleton (network mesh), causing the expanded gel to become uneven, the morphological stability during such expansion can be improved by providing absorbent layers.

Making the distribution of the superabsorbent resin in the material loose on the side on contact with the body, compact in the intermediate layer, and loose again on the side farthest from the body results in the following advantages, among others. A) This will prevent the formation of gel blocks on the side in contact with the body, and will improve the absorption capacity. B) The side in contact with the body and the side farthest from the body will act as two urine reservoirs, allowing large amounts of urine to be held until absorbed by the resin. C) The gel in the expanded intermediate layer is prevented by the upper and lower layers (with the loose distribution of superabsorbent resin) from emerging at the surface of the absorbent material. D) The loose distribution of superabsorbent resin on the top and bottom layers of the absorbent material results in less direct contact between the equipment (such as rollers and belts) and the sticky, hydrated resin during the process in which the absorbent material is produced, thereby preventing the absorbent material from sticking to the equipment and causing problems during the manufacturing process.

The distribution of superabsorbent resin in the absorbent material, that is, the combination of fiber base densities in the fiber layers forming the fiber web, should be selected as befits the intended purpose.

The absorbent material of the present invention is described in further detail based on the manufacturing method (Figure 2). The fiber web 6 is coated by means of a nip coating roll 7 with a monomer solution 8 comprising a cross linkable monomer and one initiator of a redox polymerization initiator. The web is then sprayed with another initiator solution 9, and is then guided into a polymerization tank 10 kept at between 60 and 100°C. The polymerization initiator in the monomer solution initiates a radical polymerization reaction upon contact with the other sprayed initiator, the monomer solution on the fiber base polymerizes, beginning at the surface of the liquid drops, as heat is produced, and the reaction is concluded in a few seconds. The resulting superabsorbent resin 11, which is a hydrated polymer containing about 10 to 30% moisture and also containing a large amount of residual unreacted acid monomer, is then irradiated with electron beams 12 and/or UV rays 14. Since the residual unreacted acid monomer content can be efficiently lowered by adjusting the moisture to between 20 and 30% by electron beam and UV irradiation, the moisture can be dispersed by suitable means such as a rotating brush 13 between electron beam irradiation and UV irradiation.

Then, as illustrated in Figure 3, a solution of a cross linking agent with at least 2 functional groups per molecule capable of reacting with carboxylic acid groups is applied by suitable means such as a rotating brush 17 onto the hydrated polymer, which is then guided to a heating device 18 to bring about cross linking on the resin surface at 100 to 200°C, thereby enhancing the absorption performance.

The fiber web formed of at least two fiber layers in the invention should have an overall basis weight of 10 to 1000 g/m<sup>2</sup>. Less than 10 g/m<sup>2</sup> will not allow the superabsorbent resin monomer solution to adhere in the amount needed for absorbent materials in disposable diapers. More than 100 g/m<sup>2</sup> will result in poor air permeability and will be economically disadvantageous.

In the individual fiber layers forming the fiber web, the volume (cc) of the fiber layer per gram fiber base, that is, the specific volume (cc/g), should be  $0.8 \times 10^2$  to  $1.6 \times 10^2$  (cc/g). Less than  $0.8 \times 10^2$  (cc/g) will result in the formation of a film by the monomer solution in the fiber base, while more than  $1.6 \times 10^2$  (cc/g) will result in an absorbent material that is too thick and difficult to handle in subsequent processing.

The fiber web should have an air permeability of at least 10 cc/sec (100 mmH<sub>2</sub>O). At less than 10 cc/sec (100 mmH<sub>2</sub>O), the heat rapidly generated by the polymerization of the monomer will not escape, causing the rapidly heat and undergo abnormal cross linking, which will result in the formation of poorly absorbent resin on the fibers.

The fiber base density of the fiber layers in the invention can be suitably varied depending on the type and denier of the polymer of the fiber base forming the fiber layers, as well as the basis weight and bulk, that is, specific volume, of the fiber layers themselves. The fiber layers can be readily obtained by uniformly mixing high modulus fibers and heat fusing binder fibers in a suitable proportion, forming a web using means such as carding, and then allowing the fibers to become thermally fused together. The manufacture can be readily controlled by means of the compositional ratio, combination of denier levels, and types of fiber base polymer forming the base fibers, as well as the compositional ratio and denier levels of the fiber base forming the binder fibers. The base fibers should have 2 to 20 denier and should be 32 to 128 mm long. Less than 2 denier will not result in a high compression modulus or gaps large enough to give the necessary air permeability. More than 20 denier will make it more difficult to ensure that enough monomer solution is applied. A fiber length shorter than 32 mm will result in weak interlacing between fibers, and the web will tend to come apart before the heat treatment. Longer than 128 mm will result in interlacing that is too strong, which will complicate cutting and carding.

Base fibers with such high bulk and elasticity can be readily obtained when the core is in the form of a hollow tube.

The base fibers are described below.

Using some of the base fibers in the invention to form modified cross section fibers with at least one concave part joined along the fiber axis on the fiber surface enables finer adhesion of the monomer solution on the fiber surface and thus more moderate heating during polymerization. That is, the initiator solution applied to the fiber surface is rapidly and finely dispersed through the concave parts, and the modified cross section also increases the fiber surface area, resulting in better adhesion of the monomer solution. This may be combined with adjustment of fiber base density in the present invention to modify the concentration (density) of the superabsorbent resin.

The base fibers should be at least one thermoplastic polymer selected from polyester, polyamide, polypropylene, or polyethylene fibers or the like. Polyester fibers are especially desirable because of their exceptional fiber performance.

The use of bicomponent spun fiber comprising low melting polyester polymer and polyethylene terephthalate when polyester fibers are used as the base fibers will give high bulk fibers with good air permeability because the polyester polymers will be compatible with each other when heat treated. Such low melting polyester polymers are not particularly limited, provided that they have good fiber formability, and melt and soften between 80 and 180°C. The low melting polyester polymers are preferably copolymers with terephthalic acid, isophthalic acid and ethylene glycol to facilitate the manufacture of fibers and for the sake of the fiber properties.

The binder fibers are described below.

The binder fibers should have 1.5 to 6 denier and should be 32 to 128 mm long. The use of binder fibers with low denier allows a greater number of fibers to be used, resulting in greater interlacing and connecting points, greater fiber web shape stability, and greater monomer solution adhesion, but also reduces the porosity and thus the air permeability. A denier that is too large, however, results in less interlacing and fewer connecting points, as well as a loss of fiber web bulk, compression modulus, and shape stability. At a fiber length shorter than 32 mm, the web will tend to come apart before the heat treatment. A fiber length over 128 mm will complicate cutting and carding. The binder fiber should account for between 5 and 50 wt% of the fiber web. Less than 5 wt% will result in unsatisfactory fiber web bulk and shape retention, while more than 50 wt% will fail to provide a fiber with a high porosity and good air permeability as a result of a thin, hard fiber web hand.

The binder fiber is preferably a bicomponent spun fiber comprising a combination of a low melting polymer component that melts and softens when heat treated and a polymer component that does not melt and soften, since the fiber web shape cannot be kept stable if all of the fiber melts when heat treated. Heat fusing bicomponent fibers may be core-sheath types, side-by-side types, or other types. The softening point of the low melting polymer component is not particularly limited, provided that it is at least 30°C lower than the softening point of the other polymer component. Specific examples of such low melting polyester polymers include, but are not limited to, numerous combinations, such as low melting polyester polymers and polyethylene



terephthalate, polyethylene and polyethylene terephthalate, polyethylene and polyamide, polyethylene and polypropylene, polypropylene and polyethylene terephthalate, polypropylene and polyamide. The binder fibers may also be formed by selecting two or more types of fibers from among such combinations. An example would be a combination of bicomponent spun fibers comprising a low melting polyester polymer and polyethylene terephthalate, and bicomponent spun fibers comprising polyethylene and polyethylene terephthalate.

In the case of core-sheath bicomponent spun fibers, the proportion of the low melting polymer sheath component and the polymer core component should be 10:90 to 90:10. A proportion under 10:90 will not permit core-sheath bicomponent spinning, while a proportion over 90:10 will result in poor fiber performance.

In the present invention, producing the binder fiber in the form of a hollow core-sheath bicomponent spun fiber having cross sections as illustrated in Figures 4(a) through (f) is extremely useful for dramatically improving the fiber web bulk and compression modulus. In Figure 4, 20 indicates the hollow component, 21 indicates the sheath polymer, and the shaded areas 22 indicate the core polymer.

The adhesion of the acrylic acid monomer solution can be improved by mixing a fiber base (in which the fiber surfaces have been treated with a hydrophilicization agent) with the fiber layers comprising the base fibers and binder fibers. Known surfactants with good moisturizing effects can be used as such a hydrophilicization agent. Examples include nonionic surfactants such as polyoxyethylene alkyl ether and polyoxyethylene oxypropylene block copolymers, anionic surfactants such as fatty acid salts, alkyl naphthalenesulfonates, dialkyl succinates, alkyl sulfate esters salts, and higher alcohol sulfate ester salts, and cationic surfactants such as alkylamine salts and alkyl quaternary ammonium salts. The fiber web can be provided with durable hydrophilicity by using a fiber base coated on the surface with an oligomer containing hydrophilic groups as an alternative to surfactants with moisturizing effects.

The adhesion of the acrylic acid monomer solution can also be lowered by mixing a fiber base (treated on the surface with a water-repellent agent) with the fiber layers. Examples of such agents include emulsions of silicone monomers such as methyl hydrogen polysiloxane and/or dimethyl polysiloxane to which an organic acid salt of Zn, Sn, Pb or the like or another compound has been added as a catalyst to promote oxidation condensation. Durable water-

repellent effects can be achieved by coating the fiber base, drying it, and then allowing it to cure. Water-repellent effects can be similarly obtained when an emulsion of a fluorocarbon monomer comprising an acrylic acid fluorinated alkyl ester and a perfluoromonocarboxylic acid-chrome complex salt is applied to a fiber base, dried, and then cured. The aforementioned hydrophilicization agent or water-repellent agent should be applied in an amount of 0.05 to 2.0 wt% on the fiber base.

The fiber layers used in the invention can be formed by carding, airing, or other known techniques, or combinations thereof. Carding is preferred because it produced several advantages, including the following. 1) It will provide a fiber with substantial bulk and high compression modulus. 2) It will rapidly and efficiently provide wide sheets. 3) Two or more web sheets can be readily laminated. 4) The basis weight is readily adjusted. 5) The orientation of individual fiber bases can be adjusted by attaching a condensing roll (compression roll) or the like to the take off device (card web pick up device). 6) The resulting fiber web will be uniform.

The fiber layers can be readily laminated by setting up two or more carding machines in series and sequentially placing the fiber layers formed by the carding machines on top of each other. After the fiber layers have been laminated, a fiber web comprising two or more fiber layers unified by the heat treatment described below can be obtained.

The carding machines can be conventional roller or flat cards, etc. Figure 5 illustrates an example of a flat card. In the figure, 23 indicates a cylinder, 24 indicates a flat wire fillet. The surface speed slows down or stops, and a thick fiber layer is laid on the cylinder 23. 25 is a dish plate, 26 is a feed roller, 27 is a take in roller, 28 and 29 are doffer rollers, and 30 is a take off device. An example of such a card is the tandem master card by Hollingworth USA. The card web heat treatment can be done using a well known dry nonwoven fabric air through type dryer in which hot air passes through in the thicknesswise direction of the web. Advantages of a dry nonwoven fabric manufacturing method are that the heat fusing binder fibers (which fuse at a relatively low temperature) can be dispersed in the base fibers, and the heat treatment can be performed in a totally dry state, thereby enabling the formation of a fiber web with greater bulk and higher body (compression modulus) than when a liquid binder chemical is applied and the card web is heat treated while wet. In addition, there are no safety problems caused by chemical contact with skin. The heat treatment temperature is set to between the softening point of the low

melting polymer of the binder fibers and 80°C higher than the softening point. The air-through hot air speed is 0.2 to 3.0 m/sec, and the heat treatment time is 1 to 30 seconds.

The acrylic acid superabsorbent resin monomer solution used in the invention is described below. The monomer solution is preferably an acrylic acid or an acrylic acid-methacrylic acid mixture in the form of an aqueous solution. 20 to 95% of the total carboxyl groups may be partially neutralized with an alkali metal salt or ammonium salt. Excessive partial neutralization will make it difficult to increase the aqueous solution concentration and will thus suppress the cross linking reaction, resulting in a greater proportion of water-soluble resin, which will form a blocking gel film on the resin surface. The polymerization reaction rate will drop, and the expanded gel will be weakly alkaline, which is undesirable for safety reasons. Insufficient partial neutralization will dramatically decrease the absorbent capacity of the resin. An alkali metal hydroxide or bicarbonate, or an ammonium salt, or the like can be used to neutralize the acrylic acid monomer, although sodium hydroxide or potassium hydroxide are preferred because they are readily available for industrial purposes and because they are safe and inexpensive. Potassium hydroxide is ideal since it can increase the acrylic acid monomer aqueous solution concentration. The acrylic acid monomer solution concentration should be at least 35 wt%. The higher the concentration, the greater the degree of resin polymerization and the greater the amount of monomer adhering to the fiber web. As such, the concentration is preferably slightly lower than the saturation concentration at the temperature in use, provided that no other problems are caused.

The viscosity of the monomer solution should range between 2 and 200 cps. At a viscosity less than 2 cps, the monomer solution initially adhering to the fiber base will soon rub off, preventing the formation of a thin film in the form of a sheath around the fiber base. At a viscosity more than 200 cps, the monomer solution applied to the fiber web will form into an undesirable film.

In the absorbent material of the invention, a superabsorbent resin with a urine absorption capacity (at 0.5 psi pressure) of 15 to 45 cc per gram is fixed to a fiber web with a basis weight of 10 to 100 g/m<sup>2</sup>. Normally, absorbent material used for disposable diapers is 15 cm wide by 40 cm long (surface area of about 0.06 m<sup>2</sup>). In order to absorb and retain four to five incidents of about

50 cc infant urine at a time, the amount of the superabsorbent resin must be at least twice the weight of the fiber web.

In the present invention, a stable resin gel shape can be achieved through intermolecular cross linking by adding to the acrylic acid monomer 0.01 to 1.0 wt% cross linkable monomer with at least two double bonds per molecule copolymerizable with the acrylic acid monomer, such as ethylene glycol diacrylate or N,N'-methylbisacrylamide.

At least one compound selected from water-soluble polymers such as sodium polyacrylate or polyvinyl alcohol, polymer polysaccharides such as hydroxyethyl cellulose, or microparticulate inorganic compounds such as thixotropic colloid silica can be added as a viscosity regulator to the monomer solution in the invention to improve the adhesion of the monomer solution to the fiber base.

In the present invention, the acrylic acid superabsorbent resin monomer is made into a resin by what is referred to as redox polymerization initiation, where radicals are produced by a combination of peroxide substances and reducing substances to initiate polymerization. This method requires less radical-producing activation energy than pyrolytic radical polymerization, and allows polymerization to be conducted [illegible] at relatively low temperatures, making it ideal for producing an absorbent material by applying the monomer solution to a continuously running fiber web sheet and concluding the polymerization in an extremely short period of time (several seconds).

The aforementioned peroxide substance can be at least one compound selected from hydroperoxide compounds such as t-butyl hydroperoxide, azo compounds such as azobisisobutyronitrile, and hydrogen peroxide or potassium persulfate.

The aforementioned reducing substance can be, but is not limited to, at least one compound selected from sulfites such as sodium bisulfite, reducing organic acid compounds such as L-ascorbic acid, and salt compounds such as ferrous sulfate.

Highly water-soluble polymerization initiators are preferred. The amount used will depend on the combination of initiators or the polymerization temperature, but is usually 0.01 to 2.0 wt% relative to the monomer. Less than 0.01 wt% will not permit the polymerization to progress sufficiently, while more than 2.0 wt% will not produce any further benefits.

The acrylic acid monomer solution can be efficiently applied to the fiber web running at high speed by being scattered by rotating brushes as well as kiss coating with coating rolls. The amount applied can also be readily controlled. Nip coating rolls are ideal.

Polymerization is initiated with the addition of the initiator from above the monomer solution after the monomer solution has been applied to the fiber web, as described above. An aqueous solution of the initiator should be applied in the form of fine particles using a spraying device onto the surface of the monomer solution. Higher initiator concentrations are preferred, but the concentration should be slightly lower than the saturation concentration at certain temperatures to avoid clogging the spraying nozzles. Several spraying nozzles should be arranged to ensure uniform spraying on the fiber web as a whole. The initiator should be added in an amount of 0.01 to 2.0 wt% relative to the monomer. After the monomer solution has been applied to the fiber web, the polymerization should be brought about in a polymerization tank kept at between 60 and 100°C.

The configuration of the reaction tank is not particularly limited, but when the polymerization is concluded rapidly as the fiber web is continuously conveyed, the reaction should be carried out in a polymerization tank kept at a humidity of at least 80% and a temperature of between 60 and 100°C by hot air saturated with moisture while the fiber web, as illustrated in Figure 2, is conveyed from above to below or from below to above.

If the reaction tank temperature is below 60°C, the resulting resin will have a greater basic molecular weight and will have a greater absorption capacity, but the polymerization reaction will take longer to conclude. The temperature should therefore be at least 60°C. A temperature over 100°C will result in self cross linking polymerization, with an undesirable decrease in the absorption capacity of the resin.

Normally, during the polymerization of acrylic acid monomer, greater reaction heat is generated as the reaction progresses, and it is not uncommon for sudden spikes in temperature here and there to result in abnormal reaction, but since a bulky fiber web with good air permeability is used as the carrier of the monomer solution in the invention, moisture in the monomer solution is efficiently evaporated out of the system during the polymerization, and the elimination of the latent heat cools down the heat of the reaction, so that the temperature distribution is more uniform, preventing the problem of self cross linking caused by such local

spikes in temperature and any subsequent loss of absorption capacity. In the present invention, the polymerization reaction accompanying the rapid generation of heat is concluded extremely quickly and uniformly, but as noted above, this is possible only with the use of a bulky fiber web with good air permeability as the carrier of the monomer solution.

The monomer polymerization reaction is usually concluded in a few seconds (3 to 15 seconds), with about 90 to 95 wt% of the acid monomer being converted to superabsorbent resin. The resulting resin is a hydrated polymer containing about 20 wt% moisture as well as 5 to 10 wt% unreacted residual acid monomer. This residual acrylic acid or acrylic acid/methacrylic acid mixture in the monomer lowers the absorption capacity of the superabsorbent resin, but is also a safety problem, since it can irritate the infant's skin, causing rashes and sores. The left over monomer must therefore be reduced to safe levels by being irradiated with an electron beam and/or UV rays.

Increasing the cross linking density in the superabsorbent resin surface layer in the invention can prevent what is referred to as gel blocks, which is the formation of gel in the form of paste on the surface layer. This will allow urine to rapidly penetrate into the resin, with a dramatic increase in absorption capacity. Examples of such cross linking agents include polyglycidyl ethers such as ethylene glycol diglycidyl ether with at least two functional groups per molecule that react with carboxylic acids, polyols such as diethylene glycol, and polyamines such as ethylene diamine. When such cross linkers are added to hydrated polymers and heated, extremely uniform cross linking can be formed on the surface layer of the hydrated polymer. The extent of the mesh of the reticulated structure formed by cross linking can also be controlled to a certain extent.

The bulk (cc/g) and compression modulus (%) of the fiber web used in the invention are measured in the following manner.

To determine the bulk, a fiber web is cut into squares 10 cm on each side (the sides being aligned with the machine direction and transverse direction), four such samples are placed on top of each other alternating in the machine and transverse directions, a methyl methacrylate resin sheet and a weight are then placed on top to create a load of  $0.5 \text{ g/cm}^2$  for 10 minutes, the volume  $V_1$  (cc) of the fiber web layers is determined at that time, and  $V_1$  is divided by the previously determined weight of the fiber web layers to determine the bulk (cc/g).

To determine the compression modulus (%), a load of 35 g/cm<sup>2</sup> is placed on the fiber web layer (for which the bulk V<sub>1</sub> (cc) has been determined), after 10 minutes the volume V<sub>2</sub> (cc) is determined, and the compression modulus (%) is determined by the following equation from V<sub>1</sub> and V<sub>2</sub>.

$$\text{compression modulus (\%)} = V_2/V_1 \times 100$$

The elongation strength of the fiber web used in the invention is determined using samples cut into 15 cm long by 2.5 cm wide rectangles (long side aligned with machine and transverse direction directions [sic]). Samples are restrained at either end with chucks, and the samples are adjusted to a length of 10 cm using a tensilon. The samples are then stretched at a rate of 100%/min to determine curves relating stress and elongation. The elongation strength (g/25 mm) and elongation (%) are determined when the samples break, based on the curves.

The elongation strength and elongation of the fiber web are determined in both the machine and transverse directions.

The absorption performance, that is, the absorption factor and water retention, of the absorbent material of the invention are determined using samples prepared when material dried for 2 hours at 80°C in vacuo and conditions for 8 hours at 25°C and 60 RH% is cut into squares 10 cm on each side (the sides being aligned with the machine direction and transverse direction). Samples are first weighed {a} g. Then, as illustrated in Figure 6, samples 31 are placed in 20 cm long and 15 cm wide 250 mesh nylon fabric bags 32 and dipped in a shallow bottomed container 34 filled with a test liquid (physiological saline: 0.9 wt% NaCl) 33. After an hour, the samples 31 are taken out of the nylon bags 32. As illustrated in Figure 7, the samples are placed on a 10 mesh wire netting 35, and a methyl methacrylate resin sheet 36 and weight 37 are placed on top to provide 35 g/cm<sup>2</sup> pressure to drain the samples. The samples 31 are then taken off and weighed {b} g. The samples 31 are then put back into the nylon fabric bags 32, and the nylon bags 32 are placed against the side walls of the rotating well of a centrifugal dewatering device for 90 seconds of dewatering at 150 g centrifugal force. The samples 31 are then taken out of the nylon bags 32 and weighed {c} g.

The absorption factor and water retention are determined by the following formula from {a}, {b}, and {c}.

$$\text{absorption factor (g/g)} = \frac{\{b\} - \{a\}}{\{a\}}$$

$$\text{water retention (g/g)} = \frac{\{c\} - \{a\}}{\{a\}}$$

### Action of the Invention

The absorbent material of the present invention comprises a fiber web (formed of at least two fiber layers with different fiber base densities) coated with an acrylic acid superabsorbent resin monomer solution which is then polymerized, fixing the superabsorbent resin to the fiber base. Varying the fiber base density of the fiber layers allows the concentration (density) of the superabsorbent resin in the absorbent material to be freely modified in the thicknesswise direction of the web. Selecting a suitable resin concentration (density) distribution affords the following advantages.

- i) There is no decrease in absorption capacity due to gel blocks between resins.
- ii) Large amounts of voided urine can be temporarily held in the absorbent material until it is absorbed by the resin.
- iii) Gelled, sticky resin is prevented from separating from the absorbent material.
- iv) The resin is prevented from expanding and disrupting the fiber skeleton, causing unevenness.

### Examples

The invention is illustrated in further detail by the following examples.

#### Example 1

70 wt% base fiber consisting of polyethylene terephthalate fiber (intrinsic viscosity 0.57, 6 denier, 51 mm fiber length) and 30 wt% binder fiber comprising a core-sheath bicomponent spun fiber (4 denier, fiber length 51 mm) [A] consisting of a low melting polyester polymer (intrinsic viscosity 0.35) sheath component and a polyethylene terephthalate (intrinsic viscosity 0.57) core component, in a sheath:core volume of 1:1, were uniformly blended and cut, and were



then fed into one of two carding machines set up in series to form a web sheet [I] at a rate of 50 m/min. Separately from this, instead of the above core-sheath bicomponent spun fiber [A], binder fiber comprising a core-sheath bicomponent spun fiber (4 denier, fiber length 51 mm) [B] consisting of a high density polyethylene (MI = 20, 190°C) sheath component and a polyethylene terephthalate (intrinsic viscosity 0.57) core component, in a sheath:core volume of 1:1, was uniformly blended and cut, and was then fed into the other carding machine to form a web sheet [II] at a rate of 50 m/min.

The web sheets [I] and [II] obtained above were laminated, the resulting card web was then guided to a flat belt type of air through heat treatment device, 160°C hot air was allowed to pass through the card web for 10 seconds to melt the binder fiber and join the fibers together, forming a fiber web with good shape stability.

Table 1 shows the performance of the resulting fiber web.

#### Example 2

As the fiber web obtained in Example 1 was conveyed at a rate of 50 m/min, a 40°C monomer solution containing 1.67 wt% hydrogen peroxide relative to the monomer was applied to the fiber web resulting in a monomer weight of 200 g/m<sup>2</sup>. A 5 wt% aqueous solution of L-ascorbic acid was then sprayed onto the monomer solution in an amount of 0.34 wt% relative to the monomer, and polymerization was then immediately brought about in a polymerization tank at 80°C and 80% humidity.

The monomer solution here was obtained when N,N'-methylbisacrylamide was added as a cross linkable monomer in an amount of 0.085 wt% relative to the monomer to a partially neutralized acrylic acid monomer aqueous solution with a concentrating of 65 wt%, where 60% of the total of carboxyl groups of the acrylic acid was neutralized with potassium hydroxide.

The polymerization reaction started immediately after the application of the monomer solution to the fiber web, and the reaction was concluded in about 8 seconds as heat was generated. There was 5000 ppm residual monomer in the resulting superabsorbent resin, that is, in the acrylic acid moiety potassium salt.

Next, both sides of the fiber web were irradiated with 2 megarads (Mrad) each, for a total of 4 megarads, using acceleration electron beam radiation devices 12 and 12 facing each other as

shown in Figure 2. The resin-coated web was then sprayed with water to condition the resin to 20 wt% moisture, and both sides of the fiber web were then irradiated with 100 millijoule/cm<sup>2</sup> UV rays using UV irradiation devices 14 and 14 facing each other as shown in Figure 2, thus effectively ensuring that the unreacted monomer was completely polymerized.

Table 2 shows the performance of the resulting absorbent material.

#### Comparative Example

70 wt% base fiber consisting of polyethylene terephthalate fiber (intrinsic viscosity 0.57, 6 denier, 51 mm fiber length) and 30 wt% binder fiber comprising a core-sheath bicomponent spun fiber (4 denier, fiber length 51 mm) [A] consisting of a low melting polyester polymer (intrinsic viscosity 0.35) sheath component and a polyethylene terephthalate (intrinsic viscosity 0.57) core component, in a sheath:core volume of 1:1, were uniformly blended and cut, and were then fed into a carding machine to form a web sheet [I] at a rate of 50 m/min.

An absorbent material was otherwise obtained in the same manner as in Example 2.

Tables 1 and 2 show the performance of the resulting fiber web and absorbent material.

Table 1

	Example 1	Comparative Example 1
fiber layer [I] basis weight (g/m <sup>2</sup> )	17.5	35
fiber bulk $\rho$ (g/cm <sup>3</sup> )	$8.06 \times 10^{-3}$	$8.06 \times 10^{-3}$
base fiber	blend ratio	0.7
	denier	6
	cross section peripheral length (cm)	$8.48 \times 10^{-3}$
	surface area (cm <sup>2</sup> )	7.17
binder fiber	blend ratio	0.3
	denier	4
	cross section peripheral length (cm)	$6.38 \times 10^{-3}$
	surface area (cm <sup>2</sup> )	3.48
fiber base density (cm <sup>2</sup> )	10.65	10.65
fiber layer [II] basis weight (g/m <sup>2</sup> )	17.5	
fiber bulk $\rho$ (g/cm <sup>3</sup> )	$12.5 \times 10^{-3}$	
base fiber	blend ratio	0.7
	denier	6
	cross section peripheral length (cm)	$8.48 \times 10^{-3}$
	surface area (cm <sup>2</sup> )	11.12
binder fiber	blend ratio	0.3
	denier	4
	cross section peripheral length (cm)	8.01
	surface area (cm <sup>2</sup> )	6.752
fiber base density (cm <sup>2</sup> )	17.87	
overall fiber web basis weight (g/m <sup>2</sup> )	35	35
	bulk ( $\times 10^2$ cc/g)	1.02
	compression modulus (%)	21.2
	MD elongation strength (g/25 mm)	365
	MD elongation (%)	48
	TD elongation strength (g/25 mm)	41
	TD elongation (%)	128

Table 2

	Example 1	Comparative Example 1
Water retention (g/g)	37.4	34.2
Absorption factor (g/g)	22.3	21.5
Residual monomer (ppm)	220	170
Stickiness of absorbent material surface after absorption of test solution	A layer: virtually none B layer: some	Both layers
Superabsorbent resin distribution	(less) A layer < B layer (more)	virtually uniformly distributed

## Merits of the Invention

The absorbent material of the present invention comprises a fiber web (formed of at least two fiber layers with different fiber base densities) coated with an acrylic acid superabsorbent resin monomer solution which is then polymerized, fixing the superabsorbent resin to the fiber base. Varying the fiber base density of the fiber layers allows the concentration (density) of the superabsorbent resin in the absorbent material to be freely modified in the thicknesswise direction of the web. Selecting a suitable resin concentration (density) distribution affords the following advantages.

- i) There is no decrease in absorption capacity due to gel blocks between resins.
- ii) Large amounts of voided urine can be temporarily held in the absorbent material until it is absorbed by the resin.
- iii) Gelled, sticky resin is prevented from separating from the absorbent material.
- iv) The resin is prevented from expanding and disrupting the fiber skeleton, causing unevenness.

## 4. Brief Description of the Drawings

Figure 1 is a cross section of an example of the absorbent material in the present invention. Figure 2 illustrates an example of a device for manufacturing the absorbent material of the present invention. Figure 3 illustrates a device for a cross linker treatment used in the manufacture of the absorbent material of the present invention. Figures 4(a) through (f) are cross sections hollow core-sheath bicomponent spun fibers used in the absorbent material of the present invention. Figure 5 illustrates an example of a flat carding device. Figures 6 and 7 are schematics of devices used to determine the absorption performance of the absorbent material in the present invention. Figure 8 is a cross section of a disposable diaper. Figure 9 is a cross section of a conventional absorbent material.

1: top sheet; 2: liquid-absorbing retention layer; 3: packing sheet; 4: superabsorbent resin particles; 5: wood pulp fibers; 6: fiber web; 7: coating roll; 8: monomer solution; 9: initiator solution; 10: polymerization tank; 11: superabsorbent resin; 12: electron beam radiation device; 13: rotating brush moisture-scattering device; 14: UV radiation device; 15: heater; 16: blower; 17: rotating brush cross linker solution-scattering device; 18: heating device; 20: hollow

component; 21: sheath polymer; 22: core polymer; 23: cylinder roll; 24: top wire fillet; 25: dish plate; 26: feed roller; 27: take in roller; 28: doffer roller; 29: doffer roller; 30: take off device; 31: absorbent material sample; 32: nylon mesh bag; 33: test solution (0.9% physiological saline); 34: tray; 35: wire netting; 36: acrylic resin sheet; 37: weight; F: fiber base; A and B: fiber layers

Figure 1

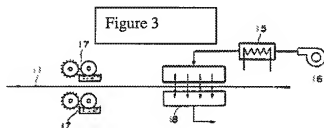
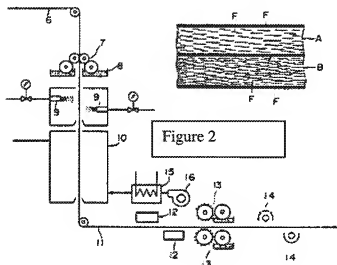


Figure 4

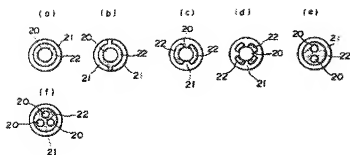


Figure 5

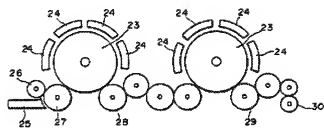


Figure 6

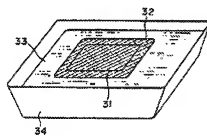


Figure 7

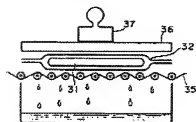


Figure 8

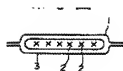
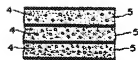


Figure 9



[Amendments to the original Japanese patent have been incorporated in the translation.]